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Rigid ceramic tile is widely used as a thermal protection system for the space shuttle program. Because rigid ceramic tile has low thermal conductivity to aerodynamic heating environment, it is being investigated for other space vehicle uses under a much severer condition. The Thermal Protection Materials Branch is currently researching and developing more-durable higher temperature ceramic tile. The initial task of this investigation is to establish a thermal and mechanical baseline of the current rigid ceramic tile. In the later tasks, newly developed ceramic tile will be compared to the established baseline for improved durability and thermal resistant.

A Thermal Diffusivity Apparatus (TDA) is used to obtain quantitative thermal conductivity of the currently used ceramic tile. This apparatus has been retrofitted to perform thermal conductivity analysis with high degree of accuracy. The TDA consists of four radiant heating elements, which are enclosed in a glass bell-jar type containment. The glass bell-jar containment, which is seated on an aluminum base plate, is piped to a reciprocating vacuum pump to achieve chamber pressure of $10E-1$ Torr. A ceramic tile, placed under the four heating elements and enclosed by the glass bell-jar, is subjected to high thermal load while under low chamber pressure.

To produce an accurate material thermal conductivity measurement, a $10E-2$ vacuum chamber environment is required to eliminate convective heat transfer. Thus, the only mode of thermal transfer from the top surface to the bottom of the ceramic tile is

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conduction. To achieve such a low chamber pressure, the aluminum base plate is now being machined to a near-perfect flat plate. This will allow a tighter seal between the glass bell-jar containment and the aluminum base plate. In addition, the replacement of existing pipe fittings, connecting from the reciprocating pump to the bell-jar, with high vacuum fittings will further reduce vacuum leakage in the system. New vacuum gauges have been incorporated to measure accurately the lower operating chamber pressure. To record quantitative thermal measurements, a data acquisition system (DAS) is now integrated to acquire in-depth and bottom-face thermocouples data of the test tile. Using Labview programming language to control the radiant heating elements and to acquire data via DAS, various thermal profiles were used to establish the thermal conductivity of the tile versus thermal heat load and flux.

Flexible ceramic insulation has been used in various space programs, and is also currently used in the shuttle program. To incorporate a rigid thermal protection system, namely rigid ceramic tile, one has to machine the tile to the contour of the space vehicle. This machining is time consuming and, therefore, costly. Flexible ceramic insulation has the benefit of fitting or contouring to the shape of the vehicle. In addition, flexible ceramic insulation is usually required thinner panel or "blanket" that offers comparable thermal resistant as to the rigid ceramic tile. Only recently, with new ceramic thread/yarn development, aerospace companies have begun the productions of flexible ceramic insulation. Flexible ceramic system is a thermal protection system that consisted of a

high temperature ceramic filler sandwiched between the two weaved ceramic fabrics. The pattern, in which the ceramic fabric is weaved, is an important factor in the system durability and thermal capability. To screen the different fabric weaved patterns, the Mini-Wind Tunnel Facility (MWTF) and the Radiant Heat Cycler (RHC) are being used to simulate and screen potential weaved pattern that can survive aerodynamic heating environment. The RHC is currently being used to subject the "blankets" to thermal load at 1 atm gauge pressure. This facility has recently incorporated with a computer controller and a data acquisition system (see Annual Progress Report June 1993 - May 1994). To reduce test turn-around time of the blankets, a testing procedure is being established to maximize the facility usage. This procedure establishes time guidelines for test article preparation, mounting to test holder, test condition setup, and test duration. The procedure also provides guidelines of setting up several test conditions at one time to maximize facility operation. In addition to facility testing procedure, a computer program is being written to do post-test analysis while test articles are being changed. Debugging the test procedure and the computer is currently in progress.

HIGH TEMPERATURE RESEARCH

Future space vehicles require much higher thermal resistant system because of the severity of the flight profiles. To answer this need, the Thermal Protection Materials Branch is developing Zr

& ZrB₂ based materials for the uses in the extreme thermal heating environment. Working with the researchers, the research students are reducing thermal data from the recent arc-jet test. Data reduction includes the production of meaningful graphs of pressure, temperature, and time versus recession measurements of the test articles. Since many composites of Zr & ZrB₂ materials were tested, determination of optimum combination of Zr & ZrB₂ will resulted from these post test graphs. In addition to arc-jet data, oxidation examination of test articles is being collected. Post test articles are prepared into an epoxy molds by the research students for sectioning and grinding. The test articles, sectioned and at various stages of grinding, revealed the level of oxidation by the microscopic examination. Measurements and photographs of each sectioned sample are collected by the research students and represented to the senior researchers for further interpretation. In addition, few post arc-jet samples are subjected to mechanical flexural testing. Research students mounted and tested various samples using an Instron for flexural characterization. Correlation of arc-jet, oxidation, and mechanical data is reported to senior researchers.

COORDINATE RESEARCH

As parts of the coordinate research, several students are working with NASA employees and contractors in the effort of characterization of new material developments. Chemical

characterization is one of many analysis that developing thermal protection materials are subjected. A useful/production ceramic tile is actually a ceramic tile with a surface protective coating. One of the more successful and commonly used coatings is known as TUF1. The production of the tile and the TUF1 coating usually takes about 65 hours, 70% of which is the production of the TUF1 coating. Recently development in coating production has reduced the production period of the TUF1 coating from +45 hrs to 9 hrs. In order to establish credential of the new production method, chemical analysis of the TUF1 from the old and new methods was needed. The old method used a ball-mill apparatus to reduce the TUF1 particles to only few microns so that it can be painted onto the uncoated ceramic tile. Because the uses of many 1/8" Dia. alumina balls in the ball-mill apparatus, alumina contaminant makes it harder to predict the amount of alumina in the final production tile. Since the amount of alumina affects the mechanical & thermal properties of tile, tile consistency is nearly impossible. With the new method, which involves Zr balls at same diameter but ball-milled at higher RPM, the desired coating size of a few microns can be achieved in just a few hours without any alumina contamination. A series of TUF1 coatings with different ball-milled time were subjected to ICP Spectrometer for chemical analysis for Zr contamination. Presently, preliminary work with alkali carbonate flux dissolution was used to dissolve TUF1 coatings for chemical analysis in the ICP Spectrometer. Research students worked closely with the other engineers to establish the data collection procedure for accurate chemical analysis of the new ball-mill procedure.

As parts of NASA Environmental Directives to keep NASA's facilities up to safety codes, research students tracked and compiled over 100 Material Safety Data Sheets (MSDS) for chemicals in various laboratories. Identification, categorization, and storage of chemicals required tremendous of time. A database of chemical type, location, and MSDS for each laboratory chemicals was established and is maintained by the research students.

Microstructure Characterization is a part of the important properties characterization that developed materials are subjected. One recently development is the usage of ceramic coatings on carbon tile and felt. Microstructure analysis was conducted using the Scanning Electronic Microscope (SEM). Research students worked side-by-side with the research engineers to determine the failure modes in mechanical and thermal testing of coated carbon tile. Coated samples are machined to 1" cubes and heat treated. After heat treatment, the research students mounted and tested the coated samples for mechanical strength as a part of the screening process. Pictures taken from SEM further screened the different coating processes and helped in determining the modes of failure of each process. An optimum coating process of the carbon tile is then determined.

Because of the uncoated ceramic tile is very porous, it has demonstrated an potential usage as artificial bone implant. In this implant, the ceramic tile aided the regeneration of the natural

bones. Because the regeneration of the nature bones is affected by the porosity of the implanted ceramic tile, determination of optimum porosity size was needed. Using SEM, different tiles (with different porosity levels) were correlated with the medical data of natural bone regeneration. Since tile is not a homogeneous material, porosity measurements varied from one part of the tile to the next. Determination of the mean porosity measurement of each sample was accomplished by the research students with the uses of SEM pictures. Different planar photographs enabled the research students to best-estimate of porosity size of the implanted samples.